52

FIRST GET-TOGETHER OF RESEARCH AND INDUSTRY

December 20–21, 1965

Petroleum Refining, Petrochemicals, Petroleum Technology and Oil & Gas Exploration

WORKING GROUP 10

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, NEW DELHI

Petroleum Refining, Petrochemicals, Petroleum Technology and Oil & Gas Exploration

WORKING GROUP 10

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH
NEW DELHI

© 1966
COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH
RAFI MARG, NEW DELHI, INDIA

Printed at Delhi Printers, 21 Daryaganj, Delhi 6

CONTENTS

Catalytic Hydrogenation of Straight-run Kerosene Distillates ... 1

S. K. Bose, A. K. Ganguli
A. N. Basu & N. G. Basak

The Case for Setting up an Indigenous Oilfield Equipment Industry ... 3

Oil India Limited, New Delhi

Manufacture of Oilfields Chemicals in India ... 8

Oil India Limited, New Delhi

Is the Need of Special Cements for Oil Well Drilling in India Overemphasized ? ... 10

F. Shikhaliev & S. N. Bhattacharya

Testing Facilities Available at IIP for Certification of Petroleum Products ... 12

J. R. Thiery & R. A. Rao

Import Substitution in Petroleum Industry ... 17

H. K. Joshi

Catalytic Hydrogenation of Straight-run Kerosene Distillates

S. K. BOSE, A. K. GANGULI, A. N. BASU & N. G. BASAK
Central Fuel Research Institute
Jealgora

One of the important criteria of assessing the quality of kerosene oils is the smoke point. It is well known that this property is very much dependent on the presence of aromatic hydrocarbons in appreciable quantities. The straight-run kerosene oil obtained from crude oil of Nahorkatiya in Assam contains appreciable quantities of aromatics and therefore has a very low smoke point (about 13 mm.).

Studies on the hydrogenation of aromatic nuclei have been summarized by Emmett. Raney nickel and Adam's platinum oxide have been found effective in the hydrogenation of organic compounds. Cobalt, rhodium, palladium, molybdenum, etc. have also been mentioned as catalysts used in the hydrogenation of aromatics. In the absence of catalyst poisons like sulphur, nitrogen and oxygen, the nickel and platinum catalysts are quite effective even at low temperatures. It was thought that a catalyst which would saturate the aromatic nuclei as well as remove the oxygen, nitrogen and sulphur in kerosene oils would be most suitable for hydrogenation to produce a kerosene oil of superior grade, having high smoke point. The normal practice for producing improved quality of kerosenes is to extract the aromatics with liquid sulphur dioxide or other organic solvents like furfural. These processes are effective no doubt, but the overall yield of the superior grade product is low, particularly with oils having appreciable quantities of aromatics. It was, therefore, thought that catalytic hydrogenation would not only produce superior grade of the product but also would effect any yield with minimum possible material loss. Investigations were, therefore, undertaken on the catalytic hydrogenation of straight-run kerosene oil distillates from the indigenous Nahorkatiya crude.

Autoclave tests of close cut fractions of Nahorkatiya crude indicated that the fraction between 170° and 300°C. could be catalytically hydrogenated for the production of quality grade kerosene.

Studies on the vapour phase hydrogenation of 170°–300°C. fraction in a continuous flow bench scale reactor using MoS_2 (6–14 mesh size) catalyst indicated that at 350°C., 1050 lb./sq. in. pressure, 1.0 as oil to catalyst space velocity and 1 :1000 as oil/tail gas ratio, a refined kerosene product of smoke point 28 mm. with a yield of 81 per cent could be produced. With increase of pressure to 1800 lb./sq. in. a product having 30 mm. smoke point could be achieved without affecting the yield.

Studies on the production of refined kerosene by furfural extraction shows that the yield of 27 mm. smoke point refined kerosene by this

process would be 68 per cent as against 81 per cent yield obtained by the hydrogenation process.

The catalyst MoS_2 is quite active for hydrorefining and retains the activity for fairly long periods with occasional regenerations.

Reduction of MoS_3 to MoS_2 can be done at very low pressure and the expected activity is obtained.

Regeneration of the used MoS_2 in situ for restoration of the original activity has to be done under pressure (1050 lb./sq. in.). Low pressure regeneration, with the other conditions being the same does not restore the original activity.

The older the MoS_2 becomes on use, the longer is the time required for its reactivation.

Judicious and gradual increase in reaction temperature during operation helps to maintain the activity of the catalyst.

The Case for Setting up an Indigenous Oilfield Equipment Industry

Oil India Limited
New Delhi

This report deals with oilfields equipment. The present state of preparedness of different industries which can undertake the manufacture of these materials and a phased programme of manufacture have been discussed in this report.

Introduction

Drilling for, production, and transportation of petroleum call for the use of a large variety of consumables and equipment, a substantial part of which has still to be imported from outside. All this calls for the expenditure, in one form or another, of a large amount of foreign exchange, and it is to the country's interest that as much of these consumables and equipment are made inside our territory as possible, consistent with tennets of safety.

Petroleum fluids are highly combustible and sometimes (when any leakage permits pre-mixing with air) explosive. Quite often, these may be under high pressure and their associated fluids can also at times be corrosive. In consequence, any failure in equipments can lead to heavy loss of life, materials and money. Sometimes, the petroleum resources of an entire pool may be placed in jeopardy. Considerations of safety and reliability are therefore treated as matters of utmost importance in any manufacture of consumables and equipments for the petroleum industry. Rigid specifications are usually laid down both for the basic ingredients and for the finished products and quality control and adherence to specifications enforced with the maximum severity.

All the above factors make oilfield equipment and machinery very specialized affairs; they cannot be purchased in the general engineering market in this country. If, due to the lack of foreign exchange, they cannot be imported, then a separate organization has to be set up within the country to manufacture them.

The initial step

The objectives discussed above can initially be achieved, without embarking on a fresh complex of factories and foundries, simply by creating a relatively small organization, the duties of which would comprise:

(a) setting up a design-cum-planning cell for oilfield equipment

(b) locating manufactures of basically allied engineering equipment, and liaising with them with a view to getting them to agree to

produce variants to suit oil industries' specifications and requirements

(c) actual designing and planning at the cell headquarters based on the arrangements proposed under (b) above

(d) farming out of orders and subsequent chasing

(e) setting up a 'pantry' as an adjunct to the 'cell', where the materials, coming forward from the various 'kitchens' under (d) above, would undergo inspection, assembly, performance testing and follow-up thereon from design/materials/manufacturing technique angles.

Once this oilfield equipment and machinery 'cell' has been rigged up, it should operate on the lines outlined above: its guiding policy should be to concentrate initially on such items as will yield the most dividends in the shortest possible time. To achieve this end, its first task will have to be: (a) to standardize on products and technology and (b) to locate indigenous manufacturing capacity both within and outside the Heavy Engineering Corporation (HEC), to guide the 'farming out'.

This 'farming out' needs a bit of elaboration. About 3 or 4 years ago, the public sector concerns, such as the HEC projects, were barely in the construction stage. Most of the acceptable private sector concerns were just initiating their re-equipment/modernization/expansion programmes: their production schedules were booked up for periods up to 2 years, and there was, therefore, little incentive for them to undertake work (for the oil industry) involving design/development work. Other factors affecting their reluctance to undertake work for the oil industry were: (a) the shortage of boiler quality steel, (b) the almost total non-availability of alloy steel, and (c) the paucity of foundries and forge shops capable of producing castings and forgings in carbon as also alloy steels: where these existed, their capacity was booked by Government contracts, particularly for railway components such as wheels, axle-boxes, couplings, etc.

The situation has since changed beyond all recognition inasmuch as: (i) the manufacturing capacity in the private sector is now many times more, both in volume and variety, than what it was 3/4 years back, (ii) the HEC's projects are just ready, and presumably about to clamour for work to keep them busy, and (iii) the steel (plain) availability position has eased off quite a bit with the commissioning of the Durgapur Steel Plant, and should be very much better in the near future with Bokaro Steel Plant coming into operation. The alloy steel availability position would also be very much better, when the Alloy Steel Project at Durgapur goes into production in the near future.

After having got the design and planning cell started, the organization should also lay on arrangements for a shop to be used as the 'pantry' mentioned earlier; here the procession of materials coming forward from the 'farming out' process would undergo one or more of the following operations: (a) inspection, (b) assembly, (c) performance testing and certification, and (d) despatch to customers or/buffer warehouses.

The 'pantry' would feed back information to the 'cell', and the two together would follow up defects from the design/materials/manufacturing technique angles, with the view to effecting improvements on the quality and cost of future products.

The possible agency

In a brochure brought out by the Heavy Engineering Corporation, it is stated that one of its projects, the Heavy Machine Building Plant, is intended, *inter alia*, to produce machinery for Heavy Oil Drilling Rigs. The description of its scope includes :

"There will be wide flexibility for the manufacture of diverse types of machinery for any heavy industry, based not only on standard designs and specifications of proved performance elsewhere but also to customers' own designs. To enable the customers have the benefit of the latest design facilities, a fully equipped Design Bureau with more than 600 Design Engineers trained in most advanced plants and industries is being established. This Bureau will be capable of undertaking various designs for any heavy industry."

Also the scope of the activities of an adjoining HEC project, the Foundry Forge Plant, is described as follows :

"In this plant, castings and forgings of practically any type and composition from a few pounds to as heavy as 100-ton piece weight will be produced. Such will be the scope of this project in the field of castings and forgings that one feels tempted to venture and say — you name it and we make it — be it an item of steel industry or equipment for mining, heavy earth moving, machine tool, power generation or for that matter anything that the country is likely to need in the near future."

From the above descriptions, there could be no better place to start the 'Design Cell' and the 'Pantry' than in the Heavy Machine Building Plant of the HEC.

As the 'cell' and the 'pantry' begin getting into their stride, the two would have to be merged and supplemented by a 'sales' or 'business' wing : the three together could be run as a separate and complete-in-itself division of the Heavy Machine Building Plant. With this condition obtaining, the design section would restrict its activities to design exclusively, leaving the 'sales' or 'business' wing to formulate and phase out manufacturing programmes, with time schedules in the context of needs and keeping in view the foreign exchange problem. As in any other commercial concern, some production features and problems, that will have to be resolved by close and continuous cooperation between all three wings, would be : (a) shop techniques and tools, (b) availability of materials, (c) delivery periods, and (d) performance guarantees regarding output, efficiency, durability, etc.

Admittedly, it will take many years before the division can attain, in respect of proficiency, parity with established manufacturers in other countries, but that exactly is all the more reason why the 'cell' should be created and got going forthwith.

The theme development

If the above is agreed, it would be better if the item, described as 'Heavy Oil Drilling Rigs' in the production programme of the Heavy Machine Building Plant were to : (a) be amended to 'Oilfield Equipment and Machinery', and (b) be brought forward for immediate implementation, rather than be deferred until the second stage.

For a start, it is suggested that the proposed Oilfield Equipment and Machinery Division (OEMD) of the Heavy Machine Building Plant

concentrates on the manufacture and/or purveying of simple but 'prolific-turnover' items such as cast steel valves, forged steel pipe fittings, quick acting unions, threaded couplings for J55 and N80 tubing, Christmas Trees and spools, landing heads, flanges and alloy steel studs, pressure vessels such as gas/oil separators and stabilizers, diaphragm type control valves, centralizers and scratchers etc. The design cell will have to establish and maintain close liaison with both ONGC and Oil India to ensure that the models produced can basically meet the requirements of both, with room possibly for minor selectivity (analogous to the seasoning in an omelette).

The division should, simultaneously with the batch manufacture of the aforementioned goods, also cater for 'custom' type orders from Oil India and the ONGC for spares and components of machinery/equipment already being operated by them, e.g. pump liners and valves, engine cylinder liners, pistons, piston rings, bearings, gears, sprockets, shafts, bushings and sleeves, etc.* Neither the crude oil producing concerns nor the private sector concerns have the design and production wherewithal to provide adequate service in this field, i.e. oilfield equipment spare parts.

It has to be borne in mind that designers, no matter whether they are engaged on the design of a new-model hydraulic coupling for a heavy oil drilling rig, or a batch of, say 24 valve seats as spare parts for Oil India's mud pumps must critically examine their selection of materials, design considerations and procedures, strength properties, processing operations, and means for ensuring adequate raw stock/interstage/finished product inspections. They will have to get down to basic mechanical engineering design principles involving, *inter alia*: (a) factors such as residual stresses, vibration, fatigue; features such as surface finish, shotpeening, cold working, nitriding, flame strengthening, considerations arising from wear, impact, high temperature, etc., (b) non-destructive methods of testing, such as magnetic particle, ultrasonic, X-ray, etc., (c) requirements of mass production, and (d) economic aspects in the selection of one method of processing over another.

A batch of 24 valve seats is not worth the while of any private sector concern, either to engage in involved design work, or to set up special tools/jigs/fixtures. It is the OEMD that must be prepared to provide this essential spare parts service for the petroleum crude producing industry.

To get the manufacture of the products mentioned above going at the proposed OEMD would take about 2/3 years. Only after this and not before, can the division afford to begin thinking about taking up the manufacture of low-turnover items such as crownblocks, swivels, tongs, elevators, travelling-blocks and hooks, rotary tables, draw-works, transmission units etc. When it does take up the manufacture of these items, the division will automatically be making a start towards the achievement of its earlier objective of making heavy oil drilling rings, inasmuch as these are the very assemblies that enter into the composition of heavy oil drilling rigs. If the aforementioned list of major assemblies to be manufactured is consolidated by mud pumps (cast steel) and odds/bits like air clutches, hydraulic couplings, etc. we will be covering almost everything in the way of general oilfield equipment (including packaged heavy oil drilling rigs), except for :

(a) I.C. Engines: These requirements could be more expeditiously met by developing/expanding the capacities of the existing manufacturers like the Kirloskar-Cummins, etc.

*After sufficient momentum and experience has been acquired, drilling bits could be taken up.

(b) Derricks and Substructures: These can easily be farmed out on specialist structural firms such as Kumardubi Engineering, Burns, Braithwaite or Jessops.

(c) Blow-out Preventers (and Control Units thereof): The manufacture of these can be taken on after everything else has been catered for.

(d) Drill Pipes: This is a very specialized item. Considering that the country would require to replace only a few strings per year, it would be advisable to import this item, for the time being at any rate. Even an industrially advanced country like the UK has not commenced manufacturing drill pipe in that country.

(e) Casing and Tubing: It is heartening to know that indigenous tube manufacturers, such as the Zenith Steel Company and the Indian Tube Company have plans to manufacture casing/tubing up to $6\frac{3}{4}$ in. diameter in J55 and N80 grade steel. When they get geared to this manufacture, which should be in about $1\frac{1}{2}$ years time from now, they should be persuaded and encouraged to undertake the manufacture of casing in the large diameters.

(f) High Pressure Water Pumps and Gas Compressors: A number of units are of immediate necessity for reservoir pressure maintenance and secondary recovery operations in the fields of Oil India Ltd. In the circumstances, there is little option but to import them from abroad, but the manufacture of spares for their operation can and should form a part of the responsibility of the proposed OEMD in due course.

Concluding remarks

It is thought that, if a really earnest start is made by the setting up of a 'cell' and 'pantry' by early 1966, and if the ramping up process is unrelentingly pushed through during the Fourth Plan period, the proposed OEMD could meet the bulk of the general oilfield equipment requirements of the country (including heavy oil drilling rigs) by about the second half of the Fifth Plan period. In this regard, as has been stressed in the preceding paragraphs, the OEMD can play a vital role. The task is a big one, calling for a good deal of organization/resourcefulness/very hard work and vigorous search (as opposed to research), but it is not an impossible one.

Once a start has been made with the OEMD, it should be the latter on which would devolve the responsibility (hitherto resting on the DGTD) for conducting studies aimed at finding out substitutes for imported finished products for the petroleum crude producing industry. This would postulate that this division would also be doing the scrutiny of applications for the import of oilfield equipment. As the volume of the division's own production and purveying is stepped up, there should be a corresponding reduction in the volume of import applications going through. Though it would take time to show results, this is the only rational way to taper off the need for foreign exchange for the purchase of oilfield equipment and essential oil well stores, without: (a) affecting the tempo of drilling and production operations in the oilfields, and (b) jeopardizing the life and future production potential of the oilfields.

Manufacture of Oilfields Chemicals in India

Oil India Limited
New Delhi

Although majority of the chemicals needed for the oilfields are already available in India, there are still important proprietary chemicals for which no substitutes are as yet available in India. This note draws attention to some of these important chemicals and suggests that investigations on the development of their substitutes and ultimately on the manufacture of the latter should be undertaken by the CSIR laboratories in liaison with the prospective manufacturers, Oil India Ltd and ONGC.

Introduction

This note deals with only the chemicals which are required for exploration and development of oilfields. Majority of the chemicals needed are available locally, but a few, nevertheless very important, are being imported or required to be imported for success in operations. These are used in drilling mud, cement and for some specialized jobs.

Drilling mud has a very important role to play in ensuring trouble-free drilling and has to be specially tailored to suit specific temperature, pressure and nature of rock. Whilst muds using indigenous ingredients have been found satisfactory for the range of conditions met in most of the wells the depths of which are not greater than 11,000 ft in drilling wells deeper than that Oil India have encountered conditions for which these ingredients are no longer adequate and for which some special chemicals like ferrochrome lignosulphonate and chrome lignite and their mixtures should be used in the mud. Although the quantities of such chemicals required for each well are not large, they play very vital roles in exploration and development of oilfield. Therefore, it is essential to carry out technological research into manufacture of these chemicals.

Ferrochrome lignosulphonate is an iron-chromium complex compound made from lignosulphonate which can be obtained from the waste of paper industry. As the paper industry is well-established in our country and there are also manufacturers of chromium compounds, there should be no difficulty in getting the raw materials. M/s India Paper Pulp Co. Ltd are manufacturing magnesium lignosulphonate in fairly large quantities and it requires some research to convert this compound or lignosulphonate obtained from any other paper mill into the above complex. The present requirement of Oil India for exploratory effort is likely to be of the order of 10 tons per annum but it may increase gradually to 40–50 tons if oil is struck in deep layers.

Lignite, the basic material for manufacture of chrome lignite, is available from several places in the country. Actually large quantities of

lignite are being mined at Neyveli and thus there should be no difficulty of raw materials. Here also research is needed to convert lignite into chromium compound. The requirement of this chemical is usually half that of ferrochrome lignosulphonate.

As in the case of drilling mud, cement which is used for bonding of the casing pipe with the wall of the well has to be tailored to meet the specific conditions. One of the important properties of cement slurry for this job is filtrate loss. As the cement slurry, i.e. a thick suspension of cement in water is pumped between the pipe and the wall, the water of the slurry filters through the porous rock and as a result the slurry may become too viscous to be pumped. Therefore any material which can reduce such filtration is a necessity. FLAC and Halad, two chemicals manufactured by Dowell and Haliburton respectively, are very effective in this respect. By using FLAC with cement, we have observed good cementation which is essential for success in production of oil and gas. The chemical nature of FLAC or Halad is not known, but they appear to be water soluble high polymers, probably polyelectrolytes. As research in the chemistry of high polymers is quite advanced in several laboratories of the country, it may be possible to undertake research on these chemicals. The present requirement of this chemical by Oil India is about 12 tons per year, but as the imported material is very costly, $ 4032/ton, it is definitely worthwhile to undertake work on this.

As development of a product similar to FLAC or Halad is likely to take some time, another product, viz. carboxymethylhydroxyethylcellulose (CMHEC) may be considered. It has been used in the Western countries to reduce filtration of water from cement slurry although it is known to be less efficient than FLAC or Halad. CMHEC is a derivative of cellulose and akin to carboxymethylcellulose; as the latter is being manufactured at two places in the country, it should not be difficult for the manufacturers to undertake manufacture of CMHEC with some effort.

Other chemicals

Several types of surfactants are used in the oil industry for the following purposes :

(i) Emulsification of oil in mud
(ii) Demulsification of crude oil
(iii) Wetting agent for wetting the rock to the fluid which is injected
(iv) Wetting agent for preferentially wetting steel with oil in aqueous media
(v) Peptizing agent for cement in oil

These are all specialized products and none of them can be put under any specific name. With the present condition of detergent industry in the country, it should be possible to develop suitable surfactants for the above objects and this can be expedited with the help of CSIR laboratories. We are in fact using Sarcol brand chemicals from M/s Sardesai Brothers for demulsification.

Therefore, in order to undertake development and ultimate manufacture of these chemicals, it is recommended that CSIR laboratories take the lead in liaison with the prospective manufacturers, Oil India and ONGC.

Is the Need of Special Cements for Oil Well Drilling in India Overemphasized ?

F. SHIKHALIEV & S. N. BHATTACHARYA
R & T Institute, ONGC
Dehra Dun

The important role played by suitable cement in oil well completion practice has been recognized since long. It is the cement which ensures bonding between the casing and the formation so as to isolate different fluid bearing formations from each other, thereby creating conditions which permit production of oil and gas from wells without complications[1].

Oil well cementation is highly specialized technique as the cement slurry has to be pumped thousands of feet below the surface into the annulus between the casing and the formations to displace drilling mud under diverse conditions of temperature, pressure and formation characteristics which, in general, adversely affect the properties of cement[2]. Thus, in each casing cementation, the choice of an appropriate cement is the foremost consideration as the cement is to respond to the desired qualities under the specific well parameters. It is, therefore, obvious that no single class of cement could be suitable for all wells whose parameters might differ widely[3].

Effective casing cementation generally demands a choice of a cement slurry with : (i) adequate specific gravity, (ii) low viscosity and water loss at low water to cement ratio, (iii) optimum thickening time, (iv) low heat of hydration, (v) low shrinkage, and (vi) adequate control on the final setting time with minimum of W.O.C. (waiting on cement) time etc.

Experience shows that a full evaluation of the qualities of the chosen cement needs to be made under simulated well conditions prior to using the cement in the well.

A careful scrutiny of the specific requirements of a cement slurry shows the apparent contradiction in its specification. Therefore, a neat portland cement slurry cannot be expected to satisfy all the requirements of a suitable oil well cement. It is, therefore, necessary to adjust the properties of a cement so as to suit the need of a particular well conditions. Adjustment of cement qualities is possible either (i) through strict control on the phase composition of the clinker during its manufacture and/or (ii) through use of additives along with the clinker or at the time of making cement slurry. Of the two controlled techniques the latter can work effectively only within a narrow limit[3]. It is scarcely possible to have wide control on a particular property using additives without impairing other desirable qualities of the cement. To illustrate this situation one can cite the action of alkali or calcium chloride on the balance of setting time of cement vis-a-vis its strength.

The problem engaged active attention of many oil companies abroad. Through extensive laboratory studies and field trials, A.P.I. has been able to formulate specifications of few types of oil well cements only. Oil well cements are now available from many foreign manufacturing companies.

Extensive drilling for establishing oil and gas reserves is being carried out in India by Oil & Natural Gas Commission and Oil India Limited. Till recently no special cement for oil well purposes was manufactured in India and well cementation was carried out with imported oil well cement or alternatively with ordinary portland cement after adjustment of their properties with suitable additives.

The limited use of ordinary portland cement in oil wells prompted the ONGC to get some special cement manufactured in India with the active collaboration of M/s Associated Cement Company. Since then the Commission has been using A.C.C. oil well cement for cementation of casing with varying degree of success. From well performance data, it appears that this cement works quite satisfactorily in wells with less complications where the bottom hole temperature is below 95°C. Only partial success results when the same cement is used in wells, with high bottom hole temperatures.

High temperature may be one of the important factors contributing for poor cementation in the wells of Nawagaon area where even at shallow depth of about 2000 metres, temperature as high as 150°C. is encountered. Such temperature is generally expected at depths of 3500–4000 metres in areas with normal geothermal gradient. Success in cementation of such high temperature wells demands a quality of cement conforming to class D A.P.I. oil well cement. A.P.I. class D cement has viscosity as low as 10 poise at low water to cement ratio and the thickening time of about 100 min. at appropriate temperature and pressure. A.C.C. oil well cement, on the other hand, shows 35–40 poise viscosity at water to cement ratio as high as 0·5. The difference in properties of the two types of cement is obvious.

Improvement in the quality of the Indian oil well cement by changing the phase composition is possibly the answer. The authors feel that it is possible to develop techniques to make different classes of oil well cements from indigenous raw materials and the developing oil industry of India will need more than one class of oil well cements for its future wells. Research on this line is needed. If research leads to the development of suitable oil well cements, it will have an important impact on the success of oilfield development and would eliminate the need felt for importing suitable cement from abroad.

REFERENCES

1. Cementing of Deep Wells, A.I. Bulatov, Moscow edition "Nedra" 1964.
2. Recent Development in the use of Bentonite in Cements, Morgan, B.E. and Dumbauld, G.K., Drilling and Production Practice, 1953, 163.
3. Portland Cements and their Application in the Oil Industry, Ludwing, N.C., Drilling and Production Practice, 1953, 183.

Testing Facilities Available at IIP for Certification of Petroleum Products

J. R. THIERY & R. A. RAO
Indian Institute of Petroleum
Dehra Dun

Engine Fuels and Lubricants

Introduction

It has been well established that the various physical, chemical and mechanical rig tests developed so far for engine fuels and lubricants are useful only for purposes of product identification and control of uniform quality in production and do not necessarily indicate the performance level of product in engine service. This is more particularly true for lubricants than fuels as the former while being used in the engines, are influenced by numerous parameters which make it difficult to simulate in any laboratory test. As such, all over the world, considerable effort has gone into development of engine tests. Many test procedures have been well standardized and all national and military standards include engine tests in the product specifications.

In India, while some engine test facilities are available for engine fuels, those for the lubricating oils are very meagre. Keeping this in mind and also the fact that proper evaluation facilities are necessarily the first requirement of any research and development organization, the Indian Institute of Petroleum (IIP) took up the establishment of such facilities and making them available for the petroleum industry and the users. Realizing that the standard engine tests, specially for lube oils, are based on foreign test methods, and expensive and involve continuous expense of foreign exchange for procurement of equipment and parts needed for the tests, the IIP already initiated programmes for the development of proper substitute methods based on indigenous equipment.

Engine test facilities at IIP

The engine test facilities available at IIP are listed as follows. The costs of tests and quantity of sample needed are given in the Appendix.

ENGINE FUELS

Gasoline anti-knock quality — This is evaluated by both ASTM Research (F-1) and Motor (F-2) methods on the standard CFR-Waukesha laboratory engine.

The road octane determination is carried out on passenger cars (Hindustan, Ambassador/Standard Herald) as per the standardized modified 'Uniontown method'. A Fiat car will also be available soon.

Diesel ignition quality — This can be evaluated by determining the cetane numbers by the ASTM (F-5) method on CFR-Waukesha lab. engine (This engine is expected to be received very shortly).

ENGINE LUBRICATING OILS

Tests to qualify diesel engine lubricating oils against MIL. L. 2104 A (U.S. specification), or current Indian draft standard, Heavy duty.

Detergency & anti-wear quality — Caterpillar 1A: This test is conducted for 480 hr, with a 0.4 per cent sulphur content fuel. It assesses the detergent and anti-wear qualities of the oil.

Petter AV1—CEC/AT4 procedure: This test is a substitute to Caterpillar 1A, widely used in Europe and included there in a great number of military and civilian specifications.

Kirloskar AV1-IIP procedure: The test methods as mentioned above being based on imported equipment and imported new spare parts (needed for each test), a substitute test method on the indigenous Kirloskar AV1 Series II engine has been developed at the IIP.

A number of tests with oils of various qualities have already been made in correlation with the Petter AV1, and repeatability of this method is now found quite satisfactory.

Limits of rating for qualification of oils against MIL.L. 2104 A/ISI Standard, heavy duty have been determined.

This method has been recently proposed to the ISI for inclusion in the Indian Standard for heavy duty motor oils.

Oxidation stability and corrosion of copper-lead bearings — CLR.L. 38 test: This test of 40 hr duration can be conducted at the IIP along the standard procedure, except for the fuel to be used in the test: this should normally be commercial iso-octane which at present must be imported and is in short supply. It is replaced at IIP by current aviation gasoline, and it is felt that this minor change would not affect much the significance of results.

Petter W1: This test has been developed in Europe as a substitute to the US CLR test, and is included there in military and civilian specifications. It is of 36 hr duration and is less costly than the CLR.

Substitute indigenous test on Villiers engine — Work is in progress to develop a substitute method on a small indigenous Villiers engine and it is expected that a test method could be standardized towards mid-1966.

Test for 'Supplement 1' level (US Military specification) *or ISI extra Heavy duty-1* — Engine tests are similar to those for MIL.L. 2104 A, but for the fuel used for detergency and anti-wear quality assessment, which is of 0.9 to 1 per cent sulphur content.

These tests can be conducted at IIP on Caterpillar 1A, or Petter AV1 engines. Substitute test on Kirloskar AV1 is under development.

Tests for 'Series 3' diesel lubricants (US specification) *or ISI extra Heavy duty-2* — The engine tests for this quality level include: the Caterpillar 1D and Caterpillar 1G, for which equipment is being received at IIP. Test methods could be standardized by late 1966.

Apart from the above two tests, CLR.L.38 as for MIL.L. 2104A, and Supplement 1, are also included.

Tests for the multipurpose motor oils '*Mil. L. 2104B*' (US latest Military specification) — The lubricants qualified against this specification are expected to give equally satisfactory service on most SI engines as well as on CI engines, except when highly supercharged. There are 4 engine tests included.

(a) Caterpillar 1H: similar to Cat, 1G. IIP is receiving the equipment for it.

(b) CLR.L. 38: Same as above.

(c) CLR. L. 43: This is a low temperature dispersancy test to assess the anti-sludge properties. Standardization of the test method at the IIP could be completed by end of 1966.

(d) Anti-rust performance test: In the United States, this test is run on 1960 Oldsmobile 8-cylinder engine in cyclic operations under light load and shut down conditions during a total period of 60 hours. A substitute test method on the CLR engine is under development in the USA and could be further developed at IIP towards 1967.

Tests for the SI engines lube oils, 'M. S.' Series — This is a civilian standard in the United States, including 5 tests references run on Oldsmobile 1958, V8 engine, Chryola 1958, V8 engine and Lincoln Mercury V8 engine.

Adoption of such tests in India is out of question.

Substitute tests for an M.S. Series adapted to European requirements is under development in Europe under the CEC.

It can be planned to develop similar tests conducted on S. I. engines most widely used in India, at a later stage. This could come as a result of the programme of work for formulation of Indian Standards for motor oils proposed by the IIP to the ISI.

Anti-wear performances of motor oils — Their assessment through conventional engine tests requires very long durations of operation and is therefore very costly.

Test methods (non-standardized) based on operation of engines fitted with radioactivated parts are currently used abroad to assess this performance.

A 'Radioactive test bench' is under installation at the IIP for a similar purpose and it is expected to be commissioned towards middle of 1966.

General engine tests — Power ratings and various operational problems can be studied on the bench for engines up to 200 BHP.

Road tests: Problems like road octane determination of motor gasolines, octane requirement of various makes of car, vapour lock formation, carburettor icing, temperature determinations etc. can be studied on the IIP's test cars, viz. one Hindustan Ambassador and one Standard Herald, at present.

Equipment for a chassis dynamometer is being received and erected. It will enable to conduct experiments as 'on the road' without being subjected to uncontrolled climatic and environmental conditions.

Operational problems of I.C. engines under altitude and extreme cold conditions: These can be studied at IIP with the help of a gold chamber for engine tests down to —40°C. (—40°F.), and a pressure reducing installation, which can simulate altitudes up to 5 to 6,000 metres.

Other Products

Transmission lubricants

Their friction reducing ability can be assessed on the 'Amsler frictiograph'.

Their extreme pressure properties, (often fixed in many standards and specifications) can be measured on the standardized 'four ball machine'.

Ball bearing greases

Their general performances can be assessed on a ball bearing testing rig under given temperatures, up to 200°C.

Their extreme pressure properties can also be assessed on the four ball machine.

APPENDIX

Cost of tests and quantity of sample

Type of product	Type of test	Available at IIP		Cost of test (Rs)	Qty of sample required (litres)
		at persent	by		
(1)	(2)	(3)	(4)	(5)	(6)
Diesel motor oil	Caterpillar 1A	Yes		5000	50
MIL.L. 2104A or ISI. H.D. type	Petter AV1 (substitute to Cat.)	do		1000	15
Certification delivered on basis of results of on one among Caterpillar or Petter AV1 or Kirloskar AV1 tests	Kirloskar AV1 (substitute to Cat./Petter, fully indigenous) CLR.L. 38	do		600	15
		do		3000	15
and one among CLR. L. 38	Petter W1 (substitute to CLR.)	do		800	15
or Petter W1 tests	Villiers (substitute to CLR/Petter W1)	—	mid-1966	500 estimated	10
Diesel Motor oil 'Supplement 1' (or ISI extra HD. 1)	Same tests as above, but with 1% sulphur content fuel	Same as above except for Kirloskar AV1	do	do	do
Diesel Motor oil 'Series III' (or ISI extra HD. 2)	Caterpillar 1D	do	do	est. 5000	50
	Caterpillar 1G		end 1966	do 5000	50
	CLR.L. 38		do	do 5000	50
MIL.L. 2104B Multipurpose motor lubricant	Caterpillar 1H		do	est. 5000	50
	CLR.L. 38		same as above		
	CLR.L. 43 Rusting Test		end 1966	est. 3000	15

(1)	(2)	(3)	(4)	(5)	(6)
Transmission lubricant/gear lubricants	*Friction test on Amsler Machine	Yes		100 to 500 (according to procedure)	2
	*E.P. test on 4 ball machine	do		70	0,2
Ball bearing crease	*Ball bearing test	do		200 to 600 (according to actual duration of test)	100 g.
	*E.P. test on 4 ball machine	do		70	50 g.
Motor gasoline	F1 and F2 octane No.	do		50 each	2
Diesel fuel	F5 cetane No.		mid-1966	80	2

*Costs for anti-wear properties of Lube motor oils on radioactive test bench, for various road tests, for general engine tests and for tests under altitude and extreme cold conditions can only be worked out specifically in each case, as these are not standardized type.

Import Substitution in Petroleum Industry

H. K. JOSHI
Esso
Bombay

This presentation is restricted to non-fuel, non-lubricant and non-petrochemical aspects of the petroleum industry, and deals with the import substitution achieved in the area and also mentions the scope for further possible import substitution. Substantial progress has already been made towards local production in this field although room exists for further progress.

The area to be covered in this paper can be broadly divided into hydrocarbon solvents, greases, speciality products and other miscellaneous uses.

Solvents

India consumes a large volume of petroleum solvents and with the growth of industries in the country, this volume is expected to increase rapidly. The solvents are used for extraction of vegetable oils from oil-cake, rubber processing, textile printing, paints as processing media for chemical reactions and as a solvent in the extraction of organic compounds from plants or other synthetic organic materials. The major volume of solvents is consumed as hexane, white spirits and special boiling spirits with a boiling range of 60 to 120°C. The actual and anticipated consumption of these three major solvents in the country during 1960–70 is given in Table 1. It can be seen from Table 1 that these solvents could represent a c.i.f. value of Rs 1.7 crores per year in 1965 rising to Rs 2.5 crores per year in 1970 if all of these solvents were imported.

Table 1 — Consumption and estimated requirement of hydrocarbon solvents

(Volume in thousands of kilolitres; Value in lakhs of rupees)

Year	Hexane		Special boiling spirit (60–120°C.)		Mineral spirits	
	Vol.	C.i.f. value	Vol.	C.i.f. value	Vol.	C.i.f. value
1960	6.0	14	25	27	27	32
1962	12.0	28	36	39	38	46
1964	17.0	39	43	48	52	64
1966	21.0	48	49	54	66	80
1968	26.0	60	57	63	78	95
1970	31.0	72	65	72	89	107

The manufacture of these solvents in the country has a double justification — first, it substitutes imports saving valuable foreign exchange which is quite significant, and secondly, it utilizes naphtha which is in surplus in the country. One of the major oil companies, i.e. Esso, was the first to visualize the above factors as early as 1960 and initiated activities for the production of two of these solvents. Their plant which consists of two superfractionators, went into production in mid-1962 and today supplies the entire needs of the country for solvent hexane. It also meets a substantial portion of the requirement of special boiling spirits with a boiling range of 60–120°C. Special mention must be made in this connection that the unit was designed entirely locally and a good portion of the fabrication and installation of the unit was also completed locally.

The unit was designed for a modest capacity of 35 kilolitres per day of hexane and 175 kilolitres per day of special boiling spirits. Having designed the unit locally the engineers concerned were able to modify this further so that today with very little additional outlay the unit has a capacity of 145 kilolitres per day of hexane and 290 kilolitres per day of special boiling spirits — almost three times the original capacity. During the period from the start of production todate over 30,000 kilolitres of hexane and over 45,000 kilolitres of special boiling spirits have been produced. The foreign exchange saving accruing to the country from this production is equivalent to a total of Rs 1.64 crores.

I may mention here that others have taken the lead given by Esso and today are planning to enter the field of manufacture of hydrocarbon solvents. Burmah-Shell is already producing special boiling spirits and today the country is self-sufficient with respect to this solvent.

As regards mineral spirits, this is consumed primarily in the Paint and Textile Printing industry. The industry has been using imported mineral spirits with a boiling range of 150–205°C. Because of this boiling range this material is almost 100 per cent kerosene, in fact a better portion of the normal kerosene, which is in short supply in the country. The actual use of this material does not really need this narrow boiling range and the high flash of the imported material. Again, taking into consideration the large surplus naphtha position in the country, it is considered in the interests of the country to modify the properties of the mineral spirits bringing them more in line with the conditions available in the country. These factors were translated into practice again first by Esso who started production on a limited scale of a solvent to substitute the imported mineral spirits. The modification was in the boiling range and flash of the material and was inspired by the desire to incorporate as much as possible of the surplus naphtha into the mineral spirits. This in turn also meant that the modified product would contain much less material in the kerosene boiling range than the imported product. First production was started in 1963 and currently the production has already reached a high figure of about 5000 kilolitres/month and can meet by and large the current demands of the country.

The introduction of this solvent from local production was very timely indeed for the paint industry since the foreign exchange crisis compelled the Government of India into banning imports of mineral spirits in 1965. In achieving such a phenomenal growth in the production capacity, certain adjustments were made into the properties of the product. Further modifications, no doubt, would have to be made to strike a

balance between the quality and volume of production. Here again other companies have shown interest in the manufacture of mineral spirits and several refineries in India are actively taking steps towards its production.

The actual volume of production by Esso todate of this solvent is in the neighbourhood of over 50,000 kilolitres and represents a foreign exchange saving of Rs 1.75 crores. As mentioned in Table 1, the anticipated demands for mineral spirits is very high and when imports are completely substituted, it represents a foreign exchange saving to the country of Rs 1 crore every year by 1970.

In addition to the above three major volume hydrocarbon solvents, a number of solvents with a comparatively small offtake are used by industry for various miscellaneous purposes mentioned earlier. Principal among these are high aromatic solvents, based on toluene, xylene, etc. and petroleum ether. Although the volume of these solvents is not high individually, the value is significant because of the high unit value. Currently it appears that economics of production would be considered borderline. However, if one takes the optimistic view that once such solvents are locally available, their demands will grow, a unit for production of these solvents may be justified. This is an area where further import substitution appears to be necessary.

Considerable growth potential exists for these solvents also in new uses like production of edible grade fish proteins from fish meal, production of coconut and other oils from fresh coconuts, etc.

Speciality oils and greases

Next to solvents, speciality oils and greases form the major areas for discussion. The speciality oils include processing oils like jute batching oil, rubber processing oil, cutting oils, textile oils, rust preventives, cable compounds, tannery oils, etc. These oils differ from each other in their boiling range, viscosity, pour point, per cent aromatics and other constituents like paraffins, naphthenes, etc. The variation in the properties makes them suitable for particular use. Until a few years back one had to use several vegetable and animal products like castor oil, turkey red oil, tallow, etc. for these uses. Substitution of these raw materials mineral oils has released these various oils for other purposes like food, etc. and have lessened the burden on agriculture.

Most important of these speciality oils is jute batching oil. This oil is used for lubricating jute fibres while weaving. The presence of the oil reduces the static electricity generated and also strengthens the fibre. This oil deserves special mention both because of its high turnover as well as due to its association with an export-oriented industry. Jute industry has the privilege of being the highest foreign exchange earner among all the items exported from this country.

The manufacture of this oil has gone through a chequered career in that the quality of the oil has been under continuous change and has had to meet the exacting demands of the United States Food and Drug Administration. Most of the jute treated with this oil is exported to the hard currency area, viz. USA and Western Europe. The industry has had to undertake continuous modification of the production facilities in order to meet the demands for quality of this oil and the industry has fully met

the challenge. Here again, Esso was the first to get into the field and start manufacture in 1960. This was followed by production at Burmah-Shell Refinery in 1961.

The total requirement of jute batching oil today is a little more than 100,000 kilolitres and is entirely met by indigenous production. The demand for this oil is expected to increase to 170,000 kilolitres by 1970. At the present rate of consumption, indigenous production is equivalent to a c.i.f. value of about Rs 1·5 crores. But for the strained relationship with Pakistan, there would be a considerable export potential for this oil.

It is estimated that the total requirement of all other speciality oils, excluding transformer oil, would be of the order of 42,000 kilolitres per year. This consists of several grades of oils with considerable variation in price. Based on an average value, the current consumption of these speciality oils represents a c.i.f. value of about Rs 2·5 crores. In this area also, a fair amount of progress has been made in local production based on imported base stocks and other chemicals. Recently, some local firms have started production of these on a comparatively small scale and one unit in Bombay is already producing medicinal grade liquid paraffin. Another unit in Bombay is regenerating spent lubricating oils. The major oil companies started local production of these oils a few years back and today are producing almost 90 per cent of their requirements. A stage has now been reached when efforts should be directed towards production of the base stocks that go into the manufacture of these speciality oils.

In the field of grease manufacture, several organizations are active. These include two major manufacturers, viz. Esso and Balmer Lawrie, along with a few smaller Indian companies. The total requirement of grease for the country today is of the order of 150,000 drums, the c.i.f. value of this quantity, if imported, would be about Rs 3 crores. It is estimated that currently almost 90 per cent of the grease requirement is met by local manufacture. However, even today imports of special types of greases continue to the tune of 3000 tons per year and it is hoped that with continuous efforts and ingenuity, even this will be produced in the country in the near future.

It must be mentioned that a great deal of effort and ingenuity has gone into the local production of both the speciality oils and greases in our effort to reduce the volume of imported components as well as to reduce the cost of manufacture. As in the case of speciality oils, even today a considerable portion of the base stocks that go into grease manufacture are imported. Further efforts must be directed towards local manufacture of these components.

One thing more that needs to be mentioned at this stage is that the quality and specifications of the imported speciality products and greases are often too good for the use concerned. They meet the rigid specifications prevalent in western countries and scientific analysis often indicates that such stringent specifications are not necessary in their usage in this country. A continuous check on these and the necessary modifications of these is one of the ways towards self-sufficiency in this field.

Pertinent information mentioned above is summarized in Table 2.

However, in this particular field, it may be mentioned that Esso has taken the lead towards indigenous production of most of the products referred to in this paper.

Table 2 — Import substitution in petroleum industry (non-fuel, non-lubricant & non-petrochemical)

(Volume in kilolitres; Value in lakhs of rupees)

Product	Industry demand (thousands)		C.i.f. value if imported		Current indigenous production		Extent of import that needs substitution	
	Current	Est. 1960	Current	Future	Volume	% of demand	Volume	Current value
SOLVENTS								
Hexane	18	31	42	72	18	100	nil	—
Special boiling spirits	45	65	51	72	45	100	nil	—
Mineral spirits	60	90	74	107	60	100	nil	—
Aromatic solvents	12	18	40	60	none	0.0	12	40
Other	5	10	20	40	none	0.0	5	10
SPECIALITY OILS								
Jute batching oil	110	171	150	233	110	100	nil	—
White oils, Cutting oils, Rust preventives, Textile oils, Rubber processing oils, Other	42	64	200	300	37	90(3)	5	25
Transformer oils	15	25	99	165	nil	0.0	15	99
GREASES								
Various grades (thousands of drums)	150	250	330	550	135	90(3)	15	25
								199

NOTE — Approximately 75 per cent of base stocks and other raw materials for these still imported

Price: Rs 2.00 Sh. 4/- $ 0.60